

REMARKS

This Reply is in response to the Office Action mailed on September 1, 2006 (Office Action) and is filed along with a two-month petition for extension of time together with an authorization to charge the extension fee. Claims 1-12 were pending at the time of the Office Action.

In this Reply, claims 1, 4, 9 and 12 have been amended and claim 8 has been cancelled. No new matter has been added.

Claims 1-12 were rejected under 35 U.S.C. §103(a) as being unpatentable based on Margrave *et al.* (U.S. Patent No. 6,645,455; referred to by the Examiner as the "primary reference") in view of Glatkowski *et al.* (U.S. Patent No. 7,060,241).

According to the Examiner:

The primary reference discloses the basic claimed process of forming SWNT films using a suspension of the nanotubes and a surfactant in a solution which is applied to a porous polymeric (PTFE) filter membrane, the solution removed and the resultant film doped with a halogen--fluorine. See Example 1, in particular column 15, lines 1-18 and 19-22. Essentially, the primary reference fails to explicitly teach that the film is optically transparent, that the membrane is dissolved (i.e., instant claim 2) and the exact thickness, optical transmission and resistance of the film. Glatkowski et al teaches that such films would be optically transparent and it is submitted that one of ordinary skill in the art would have modified the method of the primary reference to make such a film dependent on the exact final use desired for the film. In addition, it is respectfully submitted that the parameters of thickness, transparency and resistance would also have been readily determined dependent on the exact use desired for the film. Margrave et al teaches that the film would be removed from the porous membrane and it is submitted that removal by dissolving the membrane would have been obvious as such is a conventional way of providing stand-alone films.

Applicants agree with the Examiner's characterization of Margrave. However, for reasons described in detail below, Applicants respectfully disagree with several of the Examiner's assertions relating to the asserted modification of Margrave.

However, before reviewing the cited art and the Examiner's assertion based on the cited art, Applicants will first review the claimed invention as recited in amended claim 1 (copied below to show changes).

1. (Currently amended) A method of forming optically transparent and electrically conductive single walled carbon nanotubes (SWNT) films, comprising the steps of:
  - providing a porous membrane;
  - dispersing a plurality of single walled carbon nanotubes (SWNTs) into a solution, said solution including at least one surface stabilizing agent for preventing said SWNTs from flocculating out of suspension;
  - applying said solution to said membrane, and
  - removing said solution, wherein said SWNTs are forced onto a surface of said porous membrane to form an optically transparent SWNT film comprising a plurality of interpenetrated single walled carbon nanotubes disposed on said membrane, wherein said SWNT film provides a bulk resistivity  $\leq 2 \times 10^{-3}$  Ohm-cm and at least 10% optical transmission throughout a wavelength range from 0.4  $\mu$ m to 10  $\mu$ m.

Being interpenetrated, Applicants' claimed SWNT film produced by the claimed method necessarily comprises a high wt. % of SWNTs, since the film clearly needs to be primarily SWNTs for interpenetration to be possible. As will be described below, Glatkowski, which is relied on for its teaching of optical transparency, requires a low weight % SWNT/polymer composite to achieve the optical transparency. A low weight % SWNT composite clearly does not provide the claimed interpenetrated SWNT film.

Support for the term "interpenetrated" can be found in paragraphs 14 and 26 of Applicants' specification, as well as original claim 13. Support for the claimed bulk resistivity  $\leq 2 \times 10^{-3}$  Ohm-cm, can be found in several places in the specification, such as paragraph 13, copied below:

[0013] The SWNT films can be highly electrically conductive. For example, SWNT films can provide a sheet resistance of less than 200 ohm/sq, such as less than 150 ohm/sq, preferably less than 100 ohm/sq, or more preferably less than 50 ohm/sq at a thickness of no more than 100 nm.

Using the well known relation between sheet resistivity ( $R_s$ ), bulk resistivity ( $\rho$ ) and film thickness ( $t$ ):

$$R_s = \rho/t$$

solving for bulk resistivity ( $\rho$ ),  $\rho = R_s t$ .

Plugging in  $R_s \leq 200$  Ohm/Sq as disclosed above for a thickness of 100 nm =  $1 \times 10^{-5}$  cm, the bulk resistivity ( $\rho$ )  $\leq 200$  Ohm/Sq  $\cdot 1 \times 10^{-5}$  cm  $\leq 2 \times 10^{-3}$  Ohm-cm.

The claimed "at least 10% optical transmission throughout the wavelength range from 0.4  $\mu$ m to 10  $\mu$ m" was previously recited in the summary as well as former claim 8, which is now cancelled.

As amended, the claimed filtration-based method thus provides an interpenetrated SWNT film that is both electrically conductive (bulk resistivity  $\leq 2 \times 10^{-3}$  Ohm-cm) and optically transparent (at least 10% optical transmission throughout a wavelength range from 0.4  $\mu$ m to 10  $\mu$ m). As well known in the art, it is non-trivial to obtain a film (of most conductive materials) that is *both* optically transparent and highly electrically conductive because very thin films of good conductors, while transparent, are typically resistive (poorly-conductive), while thick films although being electrically conductive, are opaque (non-transparent).

Thus, for example, ultra-thin films of gold are known to be transparent, but poor electrical conductors, while thick films of gold though highly electrically conductive are opaque. As a result, Applicants respectfully submit that the Examiner over states the case in asserting the "the parameters of thickness, transparency and resistance would also have been readily determined dependent on the exact use desired for the film". This assertion implicitly assumes it was known at the time of the present invention that an interpenetrated nanotube film could provide both transparency and conductivity simultaneously. However, the truth of this assertion could only be supported with impermissible hindsight based on Applicants' disclosure. Moreover, having a motivation for conceiving a process which is capable of forming interpenetrated SWNT films having both high electrical conductivity and optical transparency is quite different from conceiving of a new process which forms interpenetrated SWNT films

having both high electrical conductivity and optical transparency due to (i) the well known tradeoff between electrical conductivity and optical transparency, and (ii) the lack of mechanical integrity of thin nanotube films required to obtain optical transparency.

Inventor Rinzler is quite familiar with the subject matter disclosed in the cited Margrave patent (assigned to William Marsh Rice University) and related work as he came from Rice University as well. As described below, Margrave exclusively teaches making the SWNT film thick enough (10-75  $\mu$ m) to possess sufficient integrity to be able to be physically peeled away from a low stick Teflon/PTFE membrane surface. Thick SWNT films disclosed by Margrave are highly opaque due to their thickness. Thus, the method for forming SWNT films disclosed in Margrave, which although similar to the claimed method regarding use of SWNTs filtered from suspension onto the surface of a filtration membrane, is different in that the present method forms thin SWNT transparent films, while Margrave must form thick films to have sufficient mechanically integrity to be peeled away from a membrane (and are thus necessarily opaque).

Applicants' key enabling step of using a selectively chemically removable membrane was unknown prior to the present invention, for any application known to Applicants. Without a method to remove the thin SWNT film from the membrane, the idea for making a thin SWNT film on a membrane was not practical because the thin SWNT film lacked mechanical integrity to be peeled away from the membrane as taught by Margrave. Only after the present Inventors realized that if a membrane could be selectively dissolved or selectively eaten away by some solvent (while leaving alone the SWNT film), it became possible to transfer the film to a substrate of choice. This inventive leap sent the present Inventors to filtration membrane catalogs looking at the chemical compatibility tables to see what type of membrane would have the needed small pores along with a high degree of porosity (to produce uniform films) and what solvents might be used to selectively dissolve the membrane. Applicants note that the purpose of chemical compatibility tables for the present invention is exactly the opposite of what such tables are designed for, i.e. to avoid dissolving the membrane by filtering the wrong solvent through them.

Turning now to the cited art, as will be explained below, Glatkowski's process is not combinable with Margrave's process, and even if combinable, the resulting film would not be Applicants' claimed interpenetrated SWNT film formed by the claimed method.

Margrave's only teaching for providing a stand alone SWNT film is a peel off technique to form the freestanding SWNT film. For example, Example 1 of Margrave discloses filtration of a nanotube comprising solution using a PTFE filter membrane (copied below):

Example 1

1.1 Fluorination of Single-Wall Carbon Nanotubes

Single-walled carbon nanotubes were produced by the dual pulsed laser vaporization of Co/Ni doped graphite rods and purified by techniques described previously (Rinzler, et al., 1998). The purification product is a metastable colloidal suspension of SWNT "ropes" (bundles of hexagonally close packed tubes ranging from a few to 200 SWNT, See Thess, et al., 1996) in a 0.2% aqueous solution of Triton X-100 surfactant. Filtering the solution through a PTFE filter membrane and washing with methanol to remove residual surfactant leaves a black film on the surface. *If this layer is sufficiently thick (10-75 µm) it can be peeled off to form a free standing film or "bucky paper" of SWNT.* This form has appreciable mechanical integrity and is convenient for handling, and for electrical conductivity and Raman scattering measurements. It is the fluorination of this "bucky paper" that is described here. (italics for emphasis only)

Significantly, Margrave consistently teaches forming essentially pure nanotube films being "sufficiently thick (10-75 µm) to be able to be "peeled off to form a free standing film or "bucky paper" of SWNT". Being essentially pure nanotube films, films disclosed by Margrave are generally interpenetrated, and are thus electrically conductive films. However, being at least 10 µm thick, as clear to those having ordinary skill in the art, essentially pure nanotube films disclosed by Margrave are opaque, not optically transparent.

Glatkowski is cited by the Examiner in an attempt to make up for the admitted deficiencies of Margrave regarding optical transparency and film thickness. Glatkowski discloses electrically conductive films. According to one embodiment, the film includes a plurality of single-walled nanotubes having a particular diameter. The disclosed film is disclosed to demonstrate good conductivity and transparency. Methods of preparing the film as well as methods of its use are also disclosed.

Although several methods for forming films are disclosed by Glatkowski, the filtration method disclosed by Margrave is not mentioned. See, e.g., col. 6, lines 40-49:

Films of this invention may be easily formed and applied to a substrate such as a dispersion of nanotubes alone in solvents such as acetone, water, ethers, and alcohols. The solvent may be removed by normal processes such as air drying, heating or reduced pressure to form the desired film of nanotubes. The films may be applied by other known processes such as spray painting, dip coating, spin coating, knife coating, kiss coating, gravure coating, screen printing, ink jet printing, pad printing, other types of printing or roll coating.

Glatkowski achieves electrical conductivity and optical transparency by using a small weight % of SWNTs in the film, such as from "0.01 to about 0.1%", which results in a good transparency" together with an electrically conductivity (and optically transmissive) generally polymeric material.

According to col. 5, lines 17 to 23:

"The instant films provide excellent conductivity and transparency at low loading of nanotubes. In a preferred embodiment, the nanotubes are present in the film at about 0.001 to about 1% based on weight. Preferably, the nanotubes are present in said film at about 0.01 to about 0.1%, which results in a good transparency and low haze".

Moreover, claims 6 and 7 recite films having low weight % SWNT.

6. The film of claim 1, wherein said nanotubes are present in said film at about 0.001 to about 1% based on weight.

7. The film of claim 1, wherein said nanotubes are present in said film at about 0.05%.

Thus, Glatkowski consistently and repeatedly teaches away from a primarily nanotube film, such as "0.001 to about 1% based on weight" nanotube film noted above to obtain "good transparency and low haze". However, as noted above, a primarily nanotube film is required to achieve the interpenetrated nanotube films taught by Margrave and claimed by Applicants. Thus, Margrave and Glatkowski teach away from their combination. According to M.P.E.P. Section 214S entitled "Consideration of Applicant's Rebuttal Arguments", "References Cannot Be

Combined Where Reference Teaches Away from Their Combination" (except below citing In re Grasselli, 713 F.2d 731, 743, 218 USPQ 769, 779 (Fed. Cir. 1983)

It is improper to combine references where the references teach away from their combination. In re Grasselli, 713 F.2d 731, 743, 218 USPQ 769, 779 (Fed. Cir. 1983) (The claimed catalyst which contained both iron and an alkali metal was not suggested by the combination of a reference which taught the interchangeability of antimony and alkali metal with the same beneficial result, combined with a reference expressly excluding antimony from, and adding iron to, a catalyst.).

Accordingly, it is improper to combine Margrave and Glatkowski.

Moreover, even assuming *arguendo* that Margrave and Glatkowski are reasonable to combine, as noted above, the asserted combination renders the Margrave method inoperable as described below (See MPEP 2143.01 and 2145 regarding inoperability) because optical transparency would not be obtained. Unlike Glatkowski, the Margrave process is a filtration process. For Margrave to achieve the optical transparency disclosed by Glatkowski as asserted by the Examiner ("it is submitted that one of ordinary skill in the art would have modified the method of the primary reference [Margrave] to make such a film dependent on the exact final use desired for the film"), the Margrave process would clearly need to be modified to use a low weight % of SWNTs (such as 0.1 to 1 wt %). However, Applicants cannot find a teaching in either Glatkowski or Margrave, or think of any method, to modify Margrave's filtration method to form Glatkowski's primarily polymer (e.g. 99 wt %) film required for optical transparency. A straight combination of Glatkowski's film components with Margrave's filtration method would lead to the polymer portion simply passing through the pores of the membrane, thus obtaining a pure polymer film rather than the desired primarily polymer (e.g. 99 wt %) film required for optical transparency as taught by Glatkowski. Thus, the asserted modification of Margrave with Glatkowski would render the film formed the same as the Margrave process itself (essentially a pure nanotube film), and thus inoperable with regard to an optically transparent film. Moreover, even assuming *arguendo* that such a method is available, the resulting film would not have interpenetrated SWNTs as claimed by Applicants due to the low wt % of SWNTs needed for high optical transparency as taught by Glatkowski.

Applicants also respectfully disagree with the Examiner's assertion that "Glatkowski et al. teaches that such films would be optically transparent". "Such films" incorrectly equates Margrave's essentially pure SWNT films with Glatkowski's films which are generally only 1 wt. % SWNT, or less, as described above.

Applicants also respectfully disagree with the Examiner's assertion that "removal by dissolving the membrane would have been obvious as such is a conventional way of providing stand alone films". Applicants were not aware at the time of the invention nor has the Examiner identified any references that teach the claimed steps of using a *filtration* (porous) membranes to form a film, then selectively removing the membrane to form a free standing film. Applicants agree that use of sacrificial layers are known (e.g. MRMS devices and that films are drop cast on other soluble surfaces), but note that if a straightforward drop casting of SWNTs from a surfactant suspension is attempted onto some non-porous surface the amount of surfactant (electrically insulating materials) left on the nanotubes impedes their intimate electrical contact and results in a resistive film. It is the porosity of Applicants' filtration membrane that permits the surfactant to be washed away.

Moreover, the present claimed invention evidences several recognized highly probative secondary indicia of nonobviousness. According to MPEP 2141, which incorporates the Graham Factual Inquiries and related case law, objective evidence of nonobviousness may include: (1) copying of the invention, (2) long felt but unsolved need for the invention, (3) failure of others to achieve the invention, (4) commercial success of the invention, (5) unexpected results created by the invention, (6) unexpected properties of the invention, (7) licenses showing industry respect for the invention, and (8) skepticism of skilled persons prior to the invention.

#### REGARDING (1): COPYING OF THE INVENTION

Following the inventor's related 2004 Science paper publication (Wu et al. Science Vol. 305, pp. 1273 (2004)) the University of Florida (the Assignee of the present invention) did a publicity release in which they quoted two respected nanotube researchers who are quoted below. This piece has since been copied throughout the World Wide Web (e.g. see <http://www.physorg.com/news1031.html>)

"Tobias Hertzel, a professor of physics and astronomy at Vanderbilt University in Nashville, said the relatively simple manufacturing process is key."

"Interest in this specific research article will most likely be directed towards the technology involved in the fabrication of thin, transparent and conductive films made of nanotubes..." he said. "Some of the features that make the work interesting for technological applications are its scalability and presumably low cost."

"Richard Martel, the Canada Research Chair on electroactive nanostructures and interfaces at the Université de Montréal in Canada, echoed Hertzel's sentiments."

"The most exciting part for me is that the technique developed by Rinzler's group is amazingly simple, and it can be scaled up to very large area substrates," he said. "I trust that this advance will find its way in applications fairly soon because it is simple, cheap and clever."

Since its publication on August 27, 2004 this Science paper describing the claimed nanotube film fabrication method has been cited 65 times based on a search recently performed. The following publications report using the claimed method to form their SWNT films (note that these do not include the Inventors' own publications which would add 7 more):

Title: Optoelectronic properties of transparent and conducting single-wall carbon nanotube thin films

Author(s): Fanchini G, Unalan HE, Chhowalla M

Source: APPLIED PHYSICS LETTERS 88 (19); Art. No. 191919 MAY 8 2006

Title: Carbon nanotube sheets as electrodes in organic light-emitting diodes

Author(s): Aguirre CM, Auvray S, Pigeon S, Izquierdo R, Desjardins P, Martel R

Source: APPLIED PHYSICS LETTERS 88 (18): Art. No. 183104 MAY 1 2006

Title: Design criteria for transparent single-wall carbon nanotube thin-film transistors

Author(s): Unalan HE, Fanchini G, Kanwal A, Du Pasquier A, Chhowalla M

Source: NANO LETTERS 6 (4): 677-682 APR 2006

Title: Selective oxidation of semiconducting single-wall carbon nanotubes by hydrogen peroxide

Author(s): Miyata Y, Maniwa Y, Kataura H

Source: JOURNAL OF PHYSICAL CHEMISTRY B 110 (1): 25-29 JAN 12 2006

Title: Conducting and transparent single-wall carbon nanotube electrodes for polymer-fullerene solar cells

Author(s): Pasquier AD, Unalan HE, Kanwal A, Miller S, Chhowalla M

Source: APPLIED PHYSICS LETTERS 87 (20): Art. No. 203511 NOV 14 2005

#### REGARDING (3) FAILURE OF OTHERS TO ACHIEVE THE INVENTION

The text below describes references relating to work that predated the Inventors' 2004

Science publication and concerns nanotube thin films. Some of these authors specifically needed transparent and conducting nanotube films, while others would have benefited from a more homogeneous, thin SWNT film (such as could be obtained using the claimed method). After most entries, Applicants have entered clarifying comments in square brackets. The first few references concern Richard E. Smalley's work with nanotube films. Smalley is a world recognized expert in nanotube films, and a co-inventor in the cited Margrave patent. Inventor Rinzler worked with Smalley at Rice and notes that very little got by Smalley, he was very interested in applications, as evidenced by his record of patenting everything; 46 issued U.S. Patent regarding carbon nanotubes as of 12/27/06). Given Smalley's work in nanotube films, and that he is co-inventor on the Margrave patent, if thin transparent and conducting films by filtration as claimed by Applicants had been obvious as asserted by the Examiner, Smalley would have done it (especially considering the resources and manpower at his disposal).

Among the other citations noted below is work by Siegmar Roth who was specifically after transparent, conductive nanotube films, but failed to come up with the claimed filtration method. Roth is a highly respected nanotube researcher at the Max Planck Institute who is one of the organizers of the worlds foremost, annual, invitation-only conferences on nanotubes (the International Winter School on the Electronic Properties of Novel Materials, IWNPEM, in Kirchberg, Austria).

References to Smalley work on films (other film references below):

"Electrochemical Tuning of Electronic Structure of Single-Walled Carbon Nanotubes: In-situ Raman and Vis-NIR Study"

Ladislav Kavan,<sup>\*,†,‡</sup> Peter Rapta,<sup>†,§</sup> Lothar Dunsch,<sup>†</sup> Michael J. Bronikowski,<sup>†,□</sup> Peter Willis,<sup>†</sup> and Richard E. Smalley, *J. Phys. Chem. B* 2001, **105**, 10764-10771

From the text:

"The working electrode was a thin film of SWCNT deposited on Pt, Au, or ITO (indium-tin oxide conducting glass). A thin film of nanotubes was deposited by spraying a freshly sonicated methanol-toluene (3:1 v/v) suspension of SWCNTs on an electrode surface heated with hot air."

"The ITO-supported SWCNTs served for in-situ vis-NIR spectroelectrochemistry..."

[Applicants' comment: So in 2001 Smalley made the transparent films to get the Vis-NIR not by a modification of Margrave (of which he is coinventor), but rather by spray coating. Note also that the films were sprayed *onto a transparent conducting electrode, ITO*, which teaches away from the idea that the nanotube films would have sufficient connectivity to themselves act as good conductors (simultaneous with their transparency) to provide the electrode.]

Magnetically aligned single wall carbon nanotube films: Preferred orientation and anisotropic transport properties

J. E. Fischer,<sup>a)</sup> W. Zhou, J. Vavro, M. C. Llaguno, C. Gathy, and R. Hagemannmueller  
M. J. Casavant, D. E. Walters,<sup>b)</sup> and R. E. Smalley, JOURNAL OF APPLIED PHYSICS  
VOLUME 93, 2157 (2003).

"Samples were prepared as described previously.<sup>5,8</sup> The starting material 12 was grown by pulsed laser ablation at 1100 °C, purified and filter deposited from suspension in the magnetic field. Robust films with strikingly obvious preferred cleavage directions were removed from the filter membranes, then annealed in vacuum at 1150 °C using a slow ramp to drive off volatiles and enhance crystallinity."

(Applicants' comment: The Smalley group discovered in 2001 that nanotubes aligned parallel to the direction of a strong magnetic field. To probe the electrical and optical properties of aligned nanotube films they built a filtration system that could go inside the bore of a high field magnet to deposit the nanotubes while they were aligned, retaining their alignment as they deposited on the membrane. The films made in 2001 were thick (opaque). As the above paper shows, as late as 2003 Smalley was still making thick films by filtration for studying magnetically aligned films. If the Margrave patent made it obvious that thin films were possible by filtration Smalley would have made thin transparent films to permit the additional probing of the anisotropy in these films by polarized Vis-NIR absorption spectroscopy. Note that theory predicted effects of anisotropy in absorption so this would have been of interest scientifically as well as commercially.)

From the Annual March American Physical Society Meeting Session C1 - Poster Session I,  
*POSTER session, Monday afternoon, March 03*  
*Room Exhibit Hall 2/3, Austin Convention Center*

[C1.014] Light Scattering Study on SWNTs Solutions

Tong Wang, Tao Liu, T. Veedu Sreekumar, Satish Kumar Rina Tannenbaum Vallerie Moore, R.H. Hauge, R.E. Smalley

SWNT/Oleum dispersions are being used to form film and fibers showing high electrical conductivity. PVP wrapped SWNTs have also been dissolved in aqueous medium. Light scattering studies have been performed in this study to understand the nanotube dispersion on both SWNT/Oleum as well as PVP/SWNT/surfactant/water dispersions. Preliminary studies on PVP wrapped SWNTs/water and SWNTs/oleum dispersion system show the existence of a critical concentration ( $c^*$ ) of SWNTs. Below  $c^*$ , the detected scattering intensity increases with concentration; and above  $c^*$ , the detected scattering intensity decreases with concentration. This observed phenomenon is attributed to the competition of scattering and strong absorption of SWNTs to visible light. Based on this study, level of SWNT dispersion is being studied. Results of these studies will be reported.

[Applicants' comment: So in 2003 Smalley was still researching alternative film fabrication strategies to Margrave]

Single-Wall Carbon Nanotube Films

T. V. Sreekumar, Tao Liu, and Satish Kumar, Lars M. Ericson, Robert H. Hauge, and Richard E. Smalley, *Chem. Mater.*, 15, 175 (2003)

Abstract:

An optically homogeneous solution/dispersion of single-wall carbon nanotubes (SWNTs) in oleum has been used to form isotropic films exhibiting fibrillar morphology. Tensile modulus, strength, and strain to failure of the film are 8 GPa, 30 MPa, and 0.5%, respectively. The electrical conductivity in the plane of the film is 1 – 105 S/m.

[Applicants' comment: See above comment]

Other transparent or thin nanotube films in the literature Prior to Inventor Rinzler, demonstrating the substantial effort and interest in homogeneous, thin, SWNT films (who, nevertheless, did not come up with the "obvious" filtration method):

Single-wall carbon nanotube\* films for photocurrent generation: a prompt response to visible-light irradiation

Barazzouk, S. <sup>a</sup>; Hotchandani, S.; Vinodgopal, K.; Kamai, P.V.

<sup>a</sup> Dept. of Chem. & Biomolecular Eng., Notre Dame Univ., IN, USA

Journal of Physical Chemistry B

Volume 108, Issue 44 , 2004, Pages 17015-17018

ISSN: 10895647, Coden: JPCBFK

Abstract

Electrophoretically deposited single-wall carbon «nanotube» (SWCNT) films on optically «transparent» electrodes are photoelectrochemically active and generate photocurrent upon visible excitation. A low photon-to-current conversion efficiency of 0.15% suggests that most of the photogenerated charge carriers are lost in the recombination process. Time-resolved transient absorption experiments confirm the charge separation following the laser-pulse excitation of SWCNTs. Relaxation of photogenerated charge carriers in the SWCNT to the lowest energy gap occurs in ~1 ps. [Journal Article; 16 Refs]

Synthesis and characterization of carbon nanotube\*-conducting polymer thin films

Ferrer-Anglada, N. <sup>a</sup>; Kaempgen, M.; Skakalova, V.; Detlaf-Weglikowska, U.; Roth, S.

<sup>a</sup> Departament de Fisica Aplicada, Univ. Politecnica de Catalunya, Barcelona, Spain Diamond and Related Materials  
Volume 13, Issue 2 , 2004, Pages 256-260  
ISSN: 09259635, Coden: DRMTE3

## Abstract

In this study heterostructures were obtained as thin films of carbon «nanotubes» (CNTs)/conducting polymers (CP) on «transparent» substrates. Single-walled carbon «nanotubes» (SWCNTs) were deposited on a «transparent» substrate and used as electrodes in order to grow a thin film of CP on it electrochemically. The resulting heterostructure may be sufficiently «transparent» and electrically conductive to be used in practical applications. We obtained different SWCNT-CP films by using polypyrrole or polyaniline as CPs, having varied throughout the chemical and electrochemical conditions. We measured the optical absorption and electrical conductivity in order to optimize the conditions for obtaining films that have both high electrical conductivity and transparency. We analyzed the thin films that were obtained by Raman spectroscopy in order to characterize them. Raman spectroscopy shows evidence of the possible interaction between CNTs and CPs. [Conference Paper; U.S. Copyright Clearance Center Code: 0925-9635/04/\$ 30.00; 17 Refs]

[Applicants' comment: See below text from this paper:]

“Our aim in this study is to obtain transparent, conductive thin films that can be deposited on a glass or plastic substrate; in the latter case the resulting heterostructured film would need to be flexible, in order to adapt it to any surface.”

### 2. Experimental procedure

#### 2.1. Samples

Very thin (near 100–150 nm) randomly oriented CNT networks, not far from their percolation threshold, were deposited on a transparent substrate: glass, quartz or plastic (PVC). The nanotubes were Single-walled carbon nanotubes (SWCNTs) grown by HiPco process. After having been prepared in a light suspension (~1% in volume) in SDS (sodium dodecyl sulfate), they were sprayed with an air-brush pistol onto the substrate. Once it had dried, the sample was submerged and shaken in pure water in order to remove the SDS. The resistances of the resulting CNT networks ranged from 0.5 to 5 kOhms”

Conducting transparent» thin films based on carbon «nanotubes» - conducting polymers, Ferrer-  
Anglada, N. <sup>a</sup>; Gomis, V.; El-Hachemi, Z.; Kaempgen, M.; Roth, S.

<sup>a</sup> Dept. de Fisica Aplicada, Univ. Politecnica de Catalunya, Spain  
AIP Conference Proceedings  
Issue 723 , 2004, Pages 591-594  
ISSN: 0094243, Coden: APCPCS

## Abstract

The present work reports on the characterization and optimization of thin «transparent» and electrically conducting films (from 120 to 180 nm thick) based on single walled carbon «nanotubes» (CNT) and conducting polymers, polypyrrole (PPy) or polyaniline (PA). We obtained a number of different CNT-PPy doped with PTS or PF<sub>6</sub> and CNT-PA under different parameters (electrodeposition time, density current or voltage) and analyzed the required properties, electrical conductivity and transparency, and other significant properties: Raman spectroscopy, and AFM, from which we can estimate the film thickness. The electrochemical conditions for the polymer thin film deposition were studied in order to improve their conductivity and transparency. Compared to the well known «transparent» conducting oxides like ITO, the best of our composite thin films are from 10 to 100 times less conductive and highly «transparent». As a great possibility, these conducting films could be prepared on a flexible substrate with a continuous deposition procedure. [Conference Paper; U.S. Copyright Clearance Center Code: 0094-243X/2004/\$ 22.00; 9 Refs]

## Water-soluble multi-walled nanotube\* and its film characteristics

Feng Wei <sup>a</sup>; Zhou Feng; Wang Xiao-Gong; Wan Mei-Xiang; Fujii Akihiko; Yoshino Katsumi  
Dept. of Chem. Eng., Tsinghua Univ., Beijing, China

Chinese Physics Letters  
Volume 20, Issue 5 , 2003, Pages 753-755  
ISSN: 0256307, Coden: CPLEEU

## Abstract

Covalent modification of multi-walled-«nanotube» (MWNT) surface-enhanced solubility in water yields a thin «transparent» shining dark-coloured film of soluble MWNT (s-MWNT) with a conductivity of 1.25 S/cm. Fourier transform infrared spectroscopy, scanning electron microscopy, transmission-electron microscopy, and UV-vis absorption spectroscopy were used for the film characterization. The result shows that enhanced interactions between s-MWNT and water and between s-MWNTs play an important role in increasing the solubility of the «nanotubes» in water and in the formation of uniform thin films. [Journal Article; 13 Refs]

## Transparent CNT composites

Kaempgen, A. <sup>a</sup>; Roth, S. <sup>a</sup> Max-Planck-Inst. für Festkörperforschung, Stuttgart, Germany  
AIP Conference Proceedings  
Issue 685 , 2003, Pages 554-558  
ISSN: 0094243, Coden: APCPCS

## Abstract

Thin networks of carbon «nanotubes» not far away from its percolation threshold are used its «transparent» electrode. At a transmission of about 90% the surface resistance is about 1 k  $\Omega$  cm and too high to replace ITO. However, it is conducting enough to be used as electrode in electrochemistry. Here the carbon «nanotube» network is used as conductive backbone structure to deposit another material. In case of conducting polymers the resistance is increasing by about 50-70% for polyaniline and polypyrrol respectively. These CNT/conducting polymer composites are still «transparent» but now more wavelength selective. The electrical properties are dominated by the CNT whereas the transmission characteristics by the polymer. [Conference Paper; U.S. Copyright Clearance Center Code: 0094-243X/03/\$ 20.00; 9 Refs]

[Applicants' comment: Roth was working hard at this, note the distinct co-authors]

#### Nanotube thin films 2004

Self-organizing high-density single-walled carbon nanotube» arrays from surfactant suspensions  
Limin Huang <sup>a</sup>; Xiaodong Cui; Dukovic, G.; O'Brien, S.P. <sup>a</sup> Dept. of Appl. Phys. & Appl. Math.,  
Columbia Univ., New York, NY, USA

#### Nanotechnology

Volume 15, Issue 11 , 2004, Pages 1450-1454

ISSN: 09574484, Coden: NNOTER

#### Abstract

Very «thin films» of oriented and densely packed single-walled carbon «nanotubes» (SWNTs) can be self-assembled on substrates from surfactant sodium dodecyl sulfate (SDS-) coated SWNT suspensions at ambient conditions. The evaporation of water causes a concentration of the SDS-coated «nanotubes» above critical micelle concentrations for SDS, and it is believed that self-organization of the SDS molecules serves as a driving force for the oriented and dense assembly of the «nanotubes.» The high degree of alignment in the SWNT «thin films» was characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM) and polarized Raman spectroscopy. [Journal Article; U.S. Copyright Clearance Center Code: 0957-4484/04/111450+05\$ 30.00; 31 Refs]

#### Macroelectronic applications of carbon nanotube» networks

Novak, J.P. ; Lay, M.D.; Perkins, F.K.; Snow, E.S. Naval Res. Lab., Washington, DC, USA  
Solid-State Electronics

Volume 48, Issue 10-11 , 2004, Pages 1753-1756

ISSN: 00381101, Coden: SSELAS

#### Abstract

We report the use of random networks of single-walled carbon «nanotubes» for electronics on large-area noncrystalline substrates. «Thin-film» transistors made from such random networks were fabricated and characterized on silicon oxide substrates. We have demonstrated both n- and p-type transistors with an effective mobility that is higher than amorphous Si or organic semiconductors. The carbon «nanotube» networks were also deposited from solution onto polymeric substrates. The ability to deposit these networks via room temperature methods defines a pathway for lightweight, unbreakable displays and other electronic applications requiring large-area, flexible substrates. [Conference Paper; U.S. Copyright Clearance Center Code: 0038-1101/04/\$ 30.00; 18 Refs]

[Applicants' comment: films grown or solution cast. Would benefit from our method.]

#### Solution casting and transfer printing single-walled carbon nanotube films

Meitl, M.A. <sup>\*</sup>; Yangxin Zhou; Gaur, A.; Seokwoo Jeon; Usrey, M.L.; Strano, M.S.; Rogers, J.A.

<sup>\*</sup> Dept. of Mater. Sci. & Eng., Illinois Univ., Urbana, IL, USA

Nano Letters

Volume 4, Issue 9 , 2004, Pages 1643-1647

ISSN: 15306984, Coden: NALEFD

#### Abstract

This paper presents methods for solution casting and transfer printing collections of individual single-walled carbon «nanotubes» (SWNTs) onto a wide range of substrates, including plastic sheets. The deposition involves introduction of a solvent that removes surfactant from a suspension of SWNTs as it is applied to a substrate. The subsequent controlled flocculation (cf) produces «films» of SWNTs with densities that can be varied between a few tubes per square micron to thick multilayers in a single deposition step and with orientation determined by the direction of solution flow. High-resolution rubber stamps inked in this manner can be used to print patterns of tubes with geometries defined by the relief structure on the surface of the stamp. «Thin film» transistors fabricated with these techniques demonstrate their potential use in flexible "macroelectronic" systems. [Journal Article; U.S. Copyright Clearance Center Code: 1530-6984/2004/\$ 27.50; 26 Refs]

[Applicants' comment: When Inventor Rinzler and Strano were both at the IWNPEM conference in 2005, Michel Strano, a co-author on this paper relayed to Inventor Rinzler how very disappointed John Rogers was when the filtration method was published in Science in 2004 since the qualities we reported were what Rogers had been working towards for years.]

#### A nonvolatile nanoelectromechanical memory element utilizing a fabric of carbon nanotubes\*

Ward, J.W.<sup>a</sup>; Meinhold, M.; Segal, B.M.; Berg, J.; Sen, R.; Sivarajan, R.; Brock, D.K.; Rueckes, T.

<sup>a</sup> Nantero, Inc., Woburn, MA, USA Proceedings. 2004 Non-Volatile Memory Technology Symposium (IEEE Cat. No.04EX947)  
2004, Pages 34-38

#### Abstract

Manufacturability of electronic devices based on carbon «nanotubes» (CNT) generally depends on the ability to manipulate and control individual structures at the molecular level. A novel technique has been developed to overcome this hurdle, allowing CNT-based nano-electromechanical devices to be fabricated directly on existing production CMOS fabrication lines. The first demonstration of this technique has resulted in a CNT nonvolatile memory element. This unique approach relies on the deposition and lithographic patterning of a 1-2 nm thick fabric of «nanotubes» which retain their molecular-scale electromechanical characteristics, even when patterned with 180 nm feature sizes. Individual patches of this CNT fabric can be elastically deformed by electro-static attraction to metal electrodes, creating a pair of stable nonvolatile states around the equilibrium of two molecular-level forces: an attractive van der Waals force and the restoring tensile strain within the deformed fabric. A CMOS-compatible fabrication process for these devices has been developed and demonstrated which is free from metallic or material contaminants and particulates. Because these nonvolatile memory elements are created in an all «thin-film» process, they can be monolithically integrated directly within existing CMOS circuitry to facilitate addressing and readout. Design considerations and preliminary device switching characteristics are presented. [Conference Paper; U.S. Copyright Clearance Center Code: 0-7803-8726-0/04/\$ 20.00; 5 Refs]

[Applicants' comment: the authors would have clearly benefited from the Applicants' claimed method]

Langmuir-Blodgett films<sup>a</sup> of single-wall carbon «nanotubes» layer-by-layer deposition and in-plane orientation of tubes

Yeji Kim<sup>a</sup>; Minami, N.; Weihong Zhu; Kazaoui, S.; Azumi, R.; Matsumoto, M.

<sup>a</sup> Nanotechnol. Res. Inst. & Collaborative Interdisciplinary Res. Team, Nat. Inst. of Adv. Ind. Sci. & Technol., Tsukuba, Japan

Japanese Journal of Applied Physics, Part 1 (Regular Papers, Short Notes & Review Papers)

Volume 42, Issue 12 , 2003, Pages 7629-7634

ISSN: 00214922, Coden: JAPNDE

#### Abstract

The Langmuir-Blodgett technique has been applied to build optically homogeneous «thin films» of chemically solubilized single-wall carbon «nanotubes» (s-SWNTs) which possess

good surface spreading properties at the air/water interface. Deposition can be performed in a layer-by-layer fashion up to 100 or more layers either by horizontal lifting or vertical dipping, allowing to readily control the «film» thickness. Their visible to near-infrared absorption spectra showing the characteristic features of semiconducting and metallic SWNTs prove the intactness of their one-dimensional electronic states during the preparation process. Polarized absorption spectroscopy and atomic force microscope (AFM) observation demonstrate that the tubes are oriented in the direction of the trough barrier (horizontal lifting) or in the dipping direction (vertical dipping). These are attributed to compression-induced or flow-induced orientation, respectively, the latter found to be much stronger than the former. The realization of homogeneous «thin films» of SWNTs with a controllable thickness and tube orientation should be an important basis for the future development of their scientific understanding and technological applications. [Journal Article; 28 Refs]

[Applicants' comments: Langmuir-Blodgett layer (LBL) methods were among the alternative film formation methods researchers were then exploring.]

Near-infrared saturable absorption of single-wall carbon nanotubes<sup>\*</sup> prepared by laser ablation method

Sakakibara, Y.<sup>†</sup>; Tatsuura, S.; Kataura, H.; Tokumoto, M.; Achiba, Y.

<sup>†</sup> Nat. Inst. of Adv. Ind. Sci. & Technol., Japan

Japanese Journal of Applied Physics, Part 2 (Letters)

Volume 42, Issue 5A , 2003, Pages L494-L496

ISSN: 00214922, Coden: JAPLD8

#### Abstract

We investigated the resonance dependence of saturable absorption (SA) properties in near-infrared in single-wall carbon «nanotube» (SWNT) materials prepared by the laser ablation method. For SWNT «thin films» coated on glass plates with a distinct single absorption band centered at 1.78  $\mu$  m, we measured SA properties using the z-scan method with just- or weak-resonance excitation using a femtosecond laser at 1.78  $\mu$  m or at 1.55  $\mu$  m, respectively. In the just-resonance experiments the laser intensities of ~50 MW/cm<sup>2</sup> induced decreases in the absorption coefficient by ~40%, but in the weak-resonance experiments one-order-higher laser intensities were necessary to obtain absorption changes of similar extents. [Journal Article; 22 Refs]

[Applicants' comment: Saturable absorbers are useful inside short pulsed laser cavities where the last thing you want to do is distort beam by putting something in that is optically inhomogeneous. Would greatly benefit from the claimed technology over the sprayed films they used.]

Homogeneous and structurally controlled thin films of single-wall carbon nanotubes by the Langmuir-Blodgett technique

Kim, Y.<sup>a</sup>; Minami, N.; Zhu, W.; Kazaoui, S.; Azumi, R.; Matsumoto, A.

Nat. Inst. of Adv. Ind. Sci. & Technol., Japan

Synthetic Metals

Volume 135-136 , 2003, Pages 747-748

ISSN: 03796779, Coden: SYMEDZ

Abstract

Optically homogeneous «thin films» of chemically solubilized single-wall carbon «nanotubes» (SWNTs) have been realized by using the Langmuir-Blodgett technique. Deposition can be performed in a layer-by-layer fashion up to 25 layers, allowing for precise control of the «film» thickness. Absorption spectra of these LB «films» preserving all the essential spectral features characteristic of semiconducting and metallic SWNTs prove that no serious alteration of their electronic structures occurred during the chemical treatment and functionalization. Polarized absorption spectroscopy demonstrates that the tubes are strongly oriented in the dipping direction. Comparison with «thin films» prepared by horizontal lifting show that both compression on the water surface and flow during vertical deposition contribute to this high degree of tube orientation. AFM profiles of SWNT monolayers indicate that most tubes are isolated or in small bundles of 1-6 nm sizes. [Conference Paper; U.S. Copyright Clearance Center Code: 0379-6779/03/\$ 30.00; 5 Refs]

[Applicant's comment: more on the alternative LBL method]

Preparation and preliminary property study of carbon nanotubes films» by electrophoretic deposition

Chunsheng Du<sup>a</sup>; Heldbrant, D.; Ning Pan

<sup>a</sup> Dept. of Biol. & Agric. Eng., California Univ., Davis, CA, USA

Materials Letters

Volume 57, Issue 2 , 2002, Pages 434-438

ISSN: 0167577, Coden: MLETDJ

Abstract

«Thin films» of carbon «nanotubes» were prepared by electrophoretic deposition (EPD) method. The «nanotubes films» were characterized by SEM and were found that the microstructure of the «films» was greatly affected by the composition of solvent used in EPD processing. The electrical surface resistance of the EPD «films» was relatively large, which possibly resulted from the hydrogen adsorption at the «nanotubes» walls during deposition process. [Journal Article; U.S. Copyright Clearance Center Code: 0167-577X/2002/\$ 22.00; 36 Refs]

[Applicants' comment: Exploring alternatives to the filtration method used by Margrave for forming nanotube films. Films deposited onto opaque electrodes so transparency not considered, nor is thickness discussed but films seem relatively thick (> 1 micron).]

### Multi-layer LB films\* of single-wall carbon «nanotubes»

Yinzhong Guo \*; Minami, N.; Kazaoui, S.; Junbiao Peng; Yoshida, M.; Miyashita, T.

\* Nat. Inst. of Adv. Ind. Sci. & Technol., Tsukuba, Japan

Physica B

Volume 323, Issue 1-4 , 2002, Pages 235-236

ISSN: 09214526, Coden: PHYBE3

#### Abstract

We propose a new processing technique of single-wall carbon «nanotubes» (SWNTs), namely, multi-layer Langmuir-Blodgett (LB) «films.» Solubilized SWNTs dispersed in an amphiphilic polymer matrix were spread on a water surface and vertically deposited on substrates. Optical absorbance at 1820 nm was perfectly proportional to the number of layers, demonstrating the successful, layer-by-layer growth of SWNT «thin films.» Moreover, polarized absorption and Raman spectra indicated a certain degree of tube orientation in the dipping direction. It was also possible to deposit LB «films» without the polymer matrix, though the «film» stability was somewhat lowered. Realization of SWNT «thin films» with precisely controlled thicknesses and optical transparency can be an important breakthrough for both basic understanding and technological applications of this novel form of carbon. [Journal Article; U.S. Copyright Clearance Center Code: 0921-4526/2002/\$ 22.00; 6 Refs]

[Applicants' comment: more on LBL method.]

### Electrochemical tuning of electronic states in single-wall carbon nanotubes\* studied by in situ absorption spectroscopy and ac resistance

Kazaoui, S. \*; Minami, N.; Matsuda, N.; Kataura, H.; Achiba, Y. Nat. Inst. of Adv. Ind. Sci. & Technol., Ibaraki, Japan

Applied Physics Letters

Volume 78, Issue 22 , 2001, Pages 3433-3435

ISSN: 00036951, Coden: APPLAB

#### Abstract

Electrochemical doping of single-wall carbon «nanotube» (SWNT) «films» and concomitant changes in their electronic states were investigated by in situ measurements of optical absorption spectra as well as of ac resistance using a nonaqueous electrolyte solution. A systematic, consistent, and reversible variation of these properties induced by the shift in the

electrode potential demonstrated the practicability of fine and continuous tuning of their electronic states. Analysis of the potential dependence of the absorbance at 0.68 eV enabled the estimation of average values of the electron affinity (4.8 eV) and the first ionization potential (5.4 eV) of semiconducting SWNTs. [Journal Article; U.S. Copyright Clearance Center Code: 0003-6951/2001/78(22)/3433(3)/\$ 18.00; 9 Refs]

[Applicants' comment: Spray coated onto transparent conducting electrode. Again teaches away from the idea that thin, transparent nanotube films would, simultaneously, be good conductors.]

### Doping mechanism in single-wall carbon nanotubes<sup>»</sup> studied by optical absorption

Jacquemin, R.<sup>2</sup>; Kazaoui, S.; Yu, D.; Hassanien, A.; Minami, N.; Kataura, H.; Achiba, Y.

<sup>2</sup> Nat. Inst. of Mater. & Chem. Res., Tsukuba, Japan

Synthetic Metals

Volume 115, Issue 1-3 , 2000, Pages 283-287

ISSN: 03796779, Coden: SYMEDZ

#### Abstract

We have separately probed the doping behavior of semiconducting and metallic single wall carbon «nanotube» (SWNT) «films,» by optical absorption and dc resistance measurements. Either electron acceptors (Br<sub>2</sub>, I<sub>2</sub>) or donors (K, Cs) were used as dopants with controlled stoichiometry. Disappearance of the absorption bands at 0.7, 1.2 (assigned to semiconducting SWNT) and 1.8 eV (assigned to metallic SWNT) and the concomitant decrease of resistance due to doping were attributed to electron depletion or filling in specific bands of semiconducting or metallic SWNT. This demonstrates the amphoteric doping behavior of SWNT. Semiconducting and metallic SWNT undergo charge transfer in a specific sequence: initially the transition at 0.7 eV, subsequently around 1.2 eV and finally the feature at 1.8 eV are affected depending on the concentration of the dopant. Changes in the electronic properties are discussed in terms of charge transfer mechanisms in the framework of the rigid-band model. [Conference Paper; U.S. Copyright Clearance Center Code: 0379-6779/2000/\$ 20.00; 16 Refs]

[Applicants' comment: sprayed film onto a transparent conducting electrode. Note that there is nothing in this early paper on thin, transparent nanotube films that discusses their electrical conductivity. The deposition onto a transparent conducting electrode, ITO, again teaches against the idea the nanotube film would itself be (simultaneously) electrically conducting. The arguments above against combination of the electrical conductivity known from Margrave, with all such prior art pertain here as well.]

### Electrochemical characterization of films<sup>»</sup> of single-walled carbon «nanotubes» and their possible application in supercapacitors

Chong-Yang Liu<sup>a</sup>; Bard, A.J.; Wudi, F.; Weitz, I.; Heath, J.R.

<sup>a</sup> Dept. of Chem. & Biochem., Texas Univ., Austin, TX, USA

Electrochemical and Solid-State Letters

Volume 2, Issue 11 , 1999, Pages 577-578

ISSN: 10990062, Coden: ESLEF6

### Abstract

«Films» of single-wall carbon «nanotubes» (SWCNTs) were cast from suspensions in several solvents on the surface of a Pt or Au electrode. Cyclic voltammetry of the «films» in MeCN did not show well-resolved waves (as distinct from «films» of C<sub>60</sub> prepared in a similar manner). However, the increase in the effective capacitance of the electrode with a SWCNT «film» at 0.5 V vs. an AgQRE was 283 F/g, which is about twice that of carbon electrodes in nonaqueous solvents. [Journal Article; 8 Refs]

### REGARDING (7) LICENSES SHOWING INDUSTRY RESPECT FOR THE INVENTION

The present invention is licensed by nRadiance LLC, which is held by NanoHoldings, LLC, as evidence by pages 1, 2 and the signature page (not numbered) executed on August 17, 2006 attached hereto as Exhibit "A". Page 2 Section 1.1.1 of the License clearly defines the licensed property as being solely the present claimed invention ["United States Patent Application 10/622,818 entitled "Transparent Electrodes from Single Wall Carbon Nanotubes"], together with "United States and foreign patent applications claiming priority" to United States Patent Application 10/622,818 recited in Section 1.1.2. As noted above, based on MPEP 2141, which incorporates the Graham Factual Inquiries and related case law, the License shows industry respect for the claimed invention, and as such represents recognized highly probative secondary indicia of nonobviousness.

Accordingly, based on the above arguments which demonstrate the inventive step and related non-obviousness of the claimed invention, together with the highly probative secondary evidence of nonobviousness including: many copying the invention, failure of many

others to achieve the invention, and a License showing industry respect for the invention, the claimed method recited in amended claim 1 which recites a filtration membrane for forming an interpenetrated SWNT film which is both electrically conductive ("bulk resistivity  $\leq 2 \times 10^{-3}$  Ohm-cm") and optically transparent (at least 10% optical transmission throughout a wavelength range from 0.4  $\mu\text{m}$  to 10  $\mu\text{m}$ ) is clearly patentable over the cited art.

Several dependent claims recited independently patentable limitations. For example, claim 2 recites the "step of separating said SWNT film from said porous membrane", while claim 3 limits claim 2 by reciting "said separating step comprises dissolving said membrane". Claim 4 recites "a thickness of said SWNT film is  $\leq 300$  nm".

Applicants believe the present application is in condition for allowance. Should the Examiner feel otherwise, Applicants request the Examiner to call the undersigned (direct line (561) 671-3662) before issuance of any office action to set up a telephonic interview to expedite the prosecution of the present application to an allowance. No fee is believed to be due other than the fee for the two-month extension of time. However, the Commissioner for Patents is hereby authorized to charge any deficiency in fees due or credit an excess in fees with the filing of the papers submitted herein during prosecution of this application to Deposit Account No. 50-0951.

Respectfully submitted,

AKERMAN SENTERFITT

Date: 11/16/07

Docket No. 5853-279

Neil R. Jetter, Registration No. 46,803  
AKERMAN SENTERFITT  
P.O. Box 3188  
West Palm Beach, FL 33402-3188  
Tel: 561-653-5000

# EXHIBIT A

Agreement No. A5540

## STANDARD EXCLUSIVE LICENSE AGREEMENT WITH SUBLICENSING TERMS

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This Agreement is made effective the 11<sup>th</sup> day of August, 2006, (the "Effective Date") by and between the University of Florida Research Foundation, Inc. (hereinafter called "UFRF"), a nonstock, nonprofit Florida corporation, and Radiance LLC (hereinafter called "Licensee"), a limited liability company organized and existing under the laws of the State of Delaware;

WHEREAS, UFRF owns certain inventions that are described in the "Licensed Patents" defined below, and certain other technology which is co-conceived and/or made by one or more employees or independent contractors of the University of Florida Board of Trustees ("Enabling Technology" as further defined below), and UFRF is willing to grant a license to Licensee under the Licensed Patents and under UFRF Intellectual Property Rights in the Enabling Technology, and Licensee desires such a license ..

NOW, THEREFORE, in consideration of the mutual covenants and agreements set forth below, the parties covenant and agree as follows:

# EXHIBIT A

## Section I Definitions

The following terms, with first letter capitalizations, shall have the meanings set forth below when used in this Agreement:

1.1 "Licensed Patents" shall refer to and mean:

### 1.1.1 Patent Applications:

United States Patent Application Serial No. 10/622,818, entitled "Transgaem Electrodes from Single Wall Carbon Nanotubes" invented by Dr. Andrew Kinsler and filed in the United States Patent Office on July 18, 2003 (USP#10937).

### 1.1.2 Other Patent Properties:

United States and foreign patent applications and patents claiming priority from one or more of the patent applications listed in Section 1.1.1 above, from one or more of the patents listed in Section 1.1.2 above, including any substitute, divisional, continuation, or continuation-in-part of such applications, and any reissues and reexaminations of the patents listed in Section 1.1.3 above and all foreign equivalents thereof.

1.2 "Licensed Product" and "Licensed Process" shall mean:

1.2.1 In the case of a Licensed Product, any product or part thereof developed by or on behalf of Licensee that:

- (a) is covered in whole or in part by an issued, unexpired claim of the Licensed Patents or pending claims of the Licensed Patents (but only so long as such claim continues to be pending), in any quantity in which such product is made, used or sold; or
- (b) employs or was discovered by Licensee employing Enabling Technology, anywhere in the world.

1.2.2 In the case of a Licensed Process:

- (a) any process which is covered in whole or in part by an issued, unexpired claim of the Licensed Patents or pending claims of the Licensed Patents (but only so long as such claim continues to be pending) in any country in which such process is practiced; or
- (b) any process which employs or was discovered by Licensee employing Enabling Technology, anywhere in the world.

1.3 "Net Sales" shall mean the monetary consideration or fair market value of non-monetary consideration (excluding consideration received for (i) investments in Licensee, (ii) research and development contracts and (iii) the acquisition or sale of Licensee, whether by merger, consolidation, sale of assets, sale of equity or otherwise) received by Licensee for the sale, license, and other disposition of Licensed Products and Licensed Processes under Licensed Patents and IPR&E Intellectual Property Rights in Enabling Technology pursuant to the terms of this Agreement, less the following deductions actually taken or given:

# EXHIBIT A

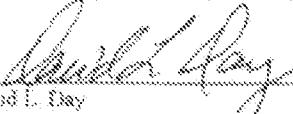
such disclosure; (g) is required by law, rule, regulation or legal process to be disclosed, provided that the receiving party making such disclosure shall take all reasonable steps to restrict and maintain to the extent possible the confidentiality of such disclosure and shall provide reasonable notice to the other party to allow such party the opportunity to oppose the required disclosure; or (h) has been independently developed by employees or others on behalf of the receiving party without access to or use of disclosing party's Confidential Information as demonstrated by written record. Each party's obligations under this Section 19 shall extend for a period of five (5) years from termination or expiration of this Agreement. It is understood and agreed that Licensee has no duty or obligation to disclose to UPRF any confidential information relating to Licensee Derivatives as described in Section 2.3 of this Agreement, or any confidential information which is not expressly and specifically identified for disclosure to UPRF in this Agreement.

## Section 20 University Rules and Regulations

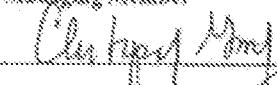
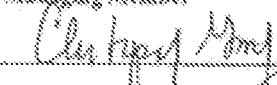
- 20.1 Licensee understands and agrees that University of Florida personnel who are engaged by Licensee, whether as consultants, employees or otherwise, or who possess a material financial interest in Licensee, are subject to the University of Florida's rule regarding outside activities and financial interests set forth in Florida Administrative Code Rule 6C1-1.011, the University of Florida's Intellectual Property Policy, and a monitoring plan which addresses conflicts of interests associated therewith.

IN WITNESS WHEREOF, the parties hereto have duly executed this Agreement on the dates indicated below.

UNIVERSITY OF FLORIDA RESEARCH FOUNDATION, INC.

  
\_\_\_\_\_  
David L. Day Date: 3/17/2006  
Director, Office of Technology Licensing

aRADIANCE LLC  
By: NanoleHoldings, LLC  
Its: Managing Member

  
\_\_\_\_\_  
By:  Date: 3/17/2006  
Name and Title: Chris Gantz, President & CEO